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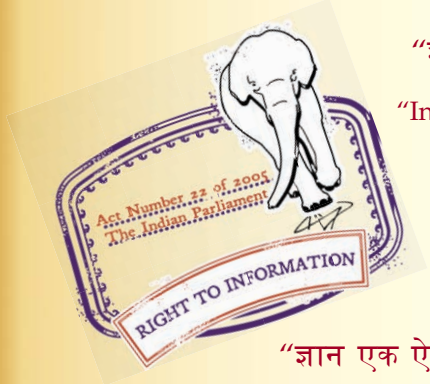
IS 56 (1993): Prussian blue (iron blue) for paints [CHD 20: Paints, Varnishes and Related Products]



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"Knowledge is such a treasure which cannot be stolen"

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IS 56 : 1993
(Reaffirmed 2004)

भारतीय मानक

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(दूसरा पुनरीक्षण)

Indian Standard

PRUSSIAN BLUE (IRON BLUE) FOR PAINTS — SPECIFICATION

(Second Revision)

UDC 667·622·117·284·4

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

July 1993

Price Group 3

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.

This standard was first published in 1950 and was largely based on the interim co-ordinated draft produced with the assistance of representatives of manufacturers and of various departments and authorities of the Government of India by the Co-ordinating Subcommittee of the No. 5 Standing Committee on Specifications for Paints and Allied Stores of the General Headquarters (now Army Headquarters), India.

In the first revision, the requirements for oil absorption and *p*H value had been modified. In this revision requirement for volatile matter has been modified and an additional requirement for residue on sieve has been added.

Due consideration has also been given to the need for alignment with ISO 2495 'Iron blue pigments for paints' published by the International Organization for Standardization (ISO) and this revised standard substantially corresponds to ISO 2495.

The composition of the committee responsible for the preparation of this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PRUSSIAN BLUE (IRON BLUE) FOR PAINTS — SPECIFICATION

(Second Revision)

1 SCOPE

1.1 This standard prescribes requirements and methods of sampling and test for prussian blue (iron blue) pigment for paints.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
33 : 1992	Methods of sampling and test for inorganic pigments and extenders for paints (*third revision*)
1070 : 1992	Reagent grade water (*third revision*)
1303 : 1983	Glossary of terms relating to paints (*second revision*)
4284 : 1967	Methods of volumetric determination of iron

3 TERMINOLOGY

3.1 For the purpose of this standard, the definitions given under **3** of IS 33 : 1992 and IS 1303 : 1983 shall apply.

4 REQUIREMENTS

4.1 Form and Condition

The material shall be in the form of fine dry powder free from grit or in such a condition that it can be readily reduced to the powder form by crushing under a palette-knife without any grinding action.

4.2 Composition

The pigment shall consist solely of the blue product formed by the reaction of solutions of iron salts with ferrocyanide or ferricyanide solution. The analysis shall show that:

a) the sum of the basic iron (as Fe) and iron cyanogen complex [expressed as Fe (CN_6)] is not less than 70 percent.

b) The total iron (expressed as Fe) is not less than 30 percent.

4.2.1 The composition of the material shall be determined as prescribed in Annex A.

4.3 Lead-Free Material

When lead free prussian blue is required, it shall contain not more than 0·03 percent of lead or compound of lead (calculated as metallic lead) when tested by the method specified under **26** of IS 33 : 1992.

4.4 The material shall also conform to the requirements given in Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be suitably packed as agreed to between the purchaser and the supplier.

5.2 Marking

The containers shall be marked with the following information:

a) Name of the material,

b) Indication of the source of manufacture,

c) Mass of the material,

d) Batch No. or lot No. in code or otherwise, and

e) Month and year of manufacture.

6 SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed under **5** of IS 33 : 1992.

7 TEST METHOD

7.1 Test shall be conducted as prescribed in IS 33 : 1992 and in Annex A. Reference to the relevant clauses of IS 33 : 1992 are given in col 4 of Table 1 and that of Annex A in **4.2.1.**

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

8 CRITERIA FOR CONFORMITY

8.1 A lot shall be declared as conforming to this standard if the test results on the composite sample satisfy the requirements prescribed under **4.**

Table 1 Requirements for Prussian Blue (Iron Blue) for Paints

(*Clause* 4.4)

Sl No.	Characteristic	Requirements	Method of Test, Ref to Cl No. in IS 33 : 1992
(1)	(2)	(3)	(4)
i)	Volatile matter, 60°C for 16 hours*, percent by mass, *Max*	4·0	8
ii)	Oil absorption	± 10 percent of approved sample	10
iii)	Colour	Close match to the approved sample	11
iv)	a) Tinting strength	Not inferior to the approved sample	13
	b) Tone	Equal to the approved sample	
v)	Matter soluble in water, percent by mass, *Max*	2·0	19
vi)	*p*H value of the aqueous extract	4·5 to 6	21
vii)	Residue on 63 micron IS Sieve percent by mass, *Max*	0·5	9

*The method given in IS 33 : 1992 (heating at 105 ± 2°C) is not suitable as the water of crystallization tends to be lost at such a high temperature and reproduceable results are not obtained. Moreover, it is a fire hazard.

ANNEX A

(*Clauses* 4.2.1 *and* 6.1)

DETERMINATION OF BASIC IRON AND IRON CYANOGEN COMPLEX

A-0 GENERAL

A-0.1 Outline of the Method

Prussian blue is decomposed by cold aqueous caustic potash. The iron cyanogen complex [$Fe(CN)_6$] forms soluble potassium ferrocyanide while the basic iron is converted into insoluble iron hydroxide which may be separated from the solution of potassium ferrocyanide by filtration.

A-1 REAGENTS

A-1.1 Standard Potassium Permanganate Solution (0·1 N)

Dissolve 3·2 g of pure potassium permanganate in 1 000 ml of water and allow it to stand for 8 to 14 days. Siphon off the clear supernatant solution into a glass stoppered bottle painted black. Weigh accurately about 3·3 g of sodium oxalate, previously dried for a few hours at 100°C and cooled over fused calcium chloride in a desiccator. Dissolve it in water and make up solution to exactly 500 ml. Transfer 25 ml of sodium oxalate to a conical flask, add 50 ml of water and 10 ml of dilute sulphuric acid (1 : 1 by volume). Heat the liquid to about 60°C and titrate with potassium permanganate solution.

$$\text{Strength of potassium permanganate solution} = \frac{M \times 25}{67 \times V}$$

where

M = mass, in g, of sodium oxalate in 1 000 ml of solution; and

V = volume in ml of potassium permanganate solution required for titration.

A-1.2 Standard Sodium Thiosulphate Solution (0·1 N)

Dissolve about 25 g of crystallized sodium thiosulphate in 1 000 ml of recently boiled water in a volumetric flask. Titrate the solution against approximately 0·1 N standard solution of iodine using starch solution as indicator.

A-1.3 Potassium Iodide

A-1.4 Saturated Solution of Sodium Bicarbonate

A-1.5 Standard Iodine Solution (0·1 N)

Dissolve in a 1 000 ml flask about 12 g of resublimed iodine in a concentrated solution of 15 to 18 g of potassium iodide. Make up the solution to 1 000 ml and standardize it against pure dry arsenious oxide. For this, dissolve about 0·25 g of arsenious oxide, accurately weighed, in the minimum quantity of hot sodium hydroxide solution. Cool and neutralize the solution with dilute hydrochloric acid, using methyl orange as indicator. Add 10 ml of sodium bicarbonate solution and dilute the solution to about 100 ml. Titrate the solution against iodine solution, using starch solution as indicator towards the end of the reaction.

A-1.6 Starch Solution

Stir up 3 g of potato starch with 10 ml of a 1-percent solution of salicylic acid and boil till starch is completely dissolved. Dilute to 1 000 ml.

A-2 PROCEDURE

A-2.1 Decomposition of Prussian Blue

Place about 0·5 g of the pigment, dried as described under **8** of IS 33 : 1992 and accurately weighed, in a 200-ml beaker. Swirling the beaker to ensure complete wetting of the material, add 10 ml of a 10 percent potassium hydroxide solution till the colour is destroyed. Filter the mixture through a large Gooch crucible, packed with prepared asbestos, and wash the residue with water. Use the residue so obtained for the determination of basic iron and the filtrate for the determination of the iron cyanogen complex.

A-2.2 Determination of Basic Iron

Extract the residue in the Gooch crucible with hot dilute hydrochloric acid and determine the iron in the solution so obtained by any suitable method (*see* IS 4284 : 1967).

A-2.2.1 If the acid extract is coloured blue, it is an indication that the residue has not been properly washed. The test shall then be repeated.

A-2.3 Determination of Iron Cyanogen Complex

A-2.3.1 Transfer the filtrate as obtained in **A-2.1** to a stoppered flask or bottle of about 1-litre capacity and dilute to 400 ml. Add 15 ml of concentrated hydrochloric acid (relative density 1·16) and 45 ml of sodium acetate solution (500 g of the pure crystallized salt to 1 litre of water) and add the permanganate solution in excess until a distinct red-brown colour is obtained and the turbidity, first formed, disappears. Add 10 ml of a 10 percent solution of potassium iodide and allow the mixture to stand for 4 minutes. Titrate the iodine liberated by the excess of potassium permanganate with standard 0·1 N sodium thiosulphate solution.

A-2.3.1.1 The quantity of potassium permanganate consumed as determined in **A-2.3.1** represents not only that necessary for the conversion of potassium ferrocyanide in potassium ferricyanide but also that necessary for the oxidation of any traces of organic matter which may be present resulting in too high a value for the iron cyanogen complex. A precise determination of the actual iron cyanogen complex can be obtained by following the procedure described in **A-2.3.2.**

A-2.3.2 Add 10 ml of hydrochloric acid (relative density 1·16), 10 ml of a 10 percent potassium iodide solution and 10 ml of zinc sulphate solution (25 g of $ZnSO_4 . 7H_2O$ in 100 ml of water) to the solution from the previous titration and allow the turbid mixture to stand for 3 minutes. Determine the liberated iodine by titration with 0·1 N sodium thiosulphate solution.

A-2.4 Calculation

Use the following factors for evaluating the results:

a) One millilitre of 0·1 N sodium thiosuphate solution is equivalent to 0·021 2 g of $Fe(CN)_6$,

b) One millilitre of 0·1 N sodium thiosulphate solution is equivalent to 0·005 585 g of iron (as Fe),

c) The sum of the basic iron (as Fe) and iron cyanogen complex [$Fe(CN)_6$] is the sum of the values determined in **A-2.2** and **A-2.3**, and

d) The total iron (as Fe) is the sum of the values determined under **A-2.2** and the value calculated for the iron cyanogen complex.

ANNEX B

(Foreword)

COMMITTEE COMPOSITION

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